NOTES

A Method of Sealing Substances in Ampullae with Inert Gases.—Usually substances susceptible to oxidation can be sealed in the vacuum of the mercury pump. However, some substances (*e. g.*, samples of vitamin-bearing extracts and the like) cannot thus be sealed, due to voluminous foaming, sputtering, etc. In such cases, the substances must be sealed in with an inert gas. There are several methods (and ampullae with two tubulures) which permit of this. The author uses a method which is almost self-explanatory from the accompanying figure, and among several other advantages permits the use of the ordinary routine ampullae with a single tubulure.

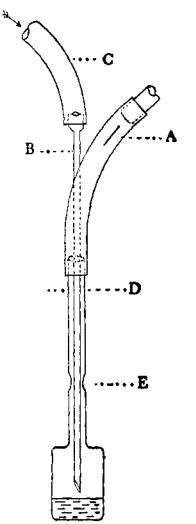

Fig. 1.

A short piece of rubber tubing is attached to the tubulure. The other end of this tubing is stoppered with a piece of glass rod, and (at A) is provided with a 1-cm. lengthwise slit, *i. e.*, a Bunsen valve. In the figure, B represents a hollow pointed surgical needle, of adequate length and lumen. The needle is passed through one wall of the tubing, and is pushed down into the ampulla almost to the surface of the substance. An inert gas (carbon dioxide or nitrogen) is conducted into the needle through tubing C, until all the air in the ampulla has been displaced and driven out through slit A. At this point the source of the inert gas is shut off, and the needle is then drawn up until its point is on a level with D. The ampulla is thereupon sealed by fusing off at E. The arrangement of Bunsen valve and needle can be used repeatedly. Due to the fact that the point of the needle can be raised or lowered, it can be adjusted to approach the surface of the substance, no matter whether large or small amounts are being sealed up. This will frequently be found to be advantageous.

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Preparation of Starch Solution for Use in Iodimetric Titrations.—Some years ago the writers and John Field, 2d [THIS JOURNAL **48,** 1299 (1926)] pointed out that clear starch solutions showing little or no Tyndall effect could be obtained for iodimetric titrations by leaching dry starch that had been ground for a long time in a pebble mill. The writers have found that equally satisfactory solutions may be obtained by leaching the

well-known breakfast food, puffed rice. The tedious grinding in the pebble mill can thus be avoided.

FROM THE FOOD RESEARCH INSTITUTE AND THE DEPARTMENT OF CHEMISTRY
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[A COMMUNICATION FROM THE LABORATORY OF ORGANIC CHEMISTRY OF THE UNIVERSITY OF WISCONSIN]

THE RATE OF HYDROGENATION OF ACETOACETIC ESTER, DEHYDROACETIC ACID, BENZENE, PHENOL AND ANILINE OVER NICKEL AT PRESSURES FROM 27 TO 350 ATMOSPHERES

BY HOMER ADKINS, HOWARD I. CRAMER AND RALPH CONNOR

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There appears to be comparatively little information available as to the relation of the pressure of hydrogen to the rate of hydrogenation of liquids with nickel catalysts. Ipatiev believed that pressures of the order of a hundred atmospheres were advantageous, while others have been but little impressed by the advantages of such pressures.[1] The experimental work described in this paper was performed in order to find out, in the case of a few supposedly representative compounds, what pressures could most advantageously be used for their hydrogenation.

The disadvantages in the use of pressures above a few atmospheres are obvious. Glass apparatus and rubber tubing may not be used, so that the assistance of a mechanic and materials often not available in the laboratory are required for the construction of the apparatus. The advantages in the use of pressures up to 80 or 150 atmospheres are no less real. Pressures of these magnitudes raise the boiling points of all organic materials which are liquids under laboratory conditions to 190° or above. This makes it possible to use any of the common solvents and to subject in the liquid phase almost all organic compounds to the action of hydrogen and the nickel catalyst. It also makes it possible to store in the reduction chamber a much greater quantity of hydrogen and thus avoid any auxiliary tank such as is necessary in reduction of pressures of a few atmospheres. This makes it possible to use a very simple set-up which includes only one inexpensive valve of the type designed for holding pressure on but one side. (Such an apparatus is manufactured by the Burgess–Parr Co. of Moline, Ill.) At least for small-scale operation in the laboratory the advantages of using pressures in the range 60 to 120 or 150 atmospheres as compared with pressures below perhaps 5 atmospheres seem to be

[1] Sabatier and Reid, "Catalysis in Organic Chemistry," D. Van Nostrand Co., New York, 1922, p. 207.